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Evaluating toxicity of Asana XL® (esfenvalerate) amendments in agricultural ditch mesocosms

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Abstract

Agricultural ditches primarily serve to remove and store excess water associated with irrigation and storm events. The ability of these ecosystems to mitigate potential contaminants is not well understood. Five sites along a 650-m agricultural ditch located in the Mississippi Delta Management Systems Evaluation Area (MDMSEA) were used to measure fate and effects of an esfenvalerate (insecticide) exposure. Following a 0.64-cm simulated storm event, samples were collected from water and sediments and analyzed spatially from five sites and temporally from 0.5 h to 56 d. Results of aqueous toxicity bioassays indicated that lethality progressed downstream throughout all sampling intervals, while sediment toxicity bioassays only elicited biological responses at the point of pesticide application to the ditch (0 m). Significant reductions in survival of *Ceriodaphnia dubia* and *Pimephales promelas* in water were measured at the 0-, 20-, and 80-m sites following application. Ten-day solid phase sediment testing of *Chironomus tentans* indicated persistent toxicity only at the point of application (0 m) and throughout 56 d (mean = 14.4% survival). No lethality or significant reduction in midge growth was measured for remaining downstream sites. These measurements were used to evaluate the potential of agricultural ditches to reduce potential deleterious effects of contaminants in agricultural drainage systems that precede receiving streams.

Keywords: Esfenvalerate; Toxicity; Agriculture; Non-point source runoff

1. Introduction

The beneficial role of wetland ecosystems for improving water quality has been established through investigating their potential for trapping and transforming contaminants associated with agricultural runoff (De Laney, 1995). Benefits of aquatic macrophytes associated with these ecosystems have also been documented in the removal of organic chemicals from the water column as well as in the reduction of suspended sediments and nutrients (Brix and Schlerup, 1989; Hand et al., 2001; Merlin et al., 2002). Recognition that agricultural drainage ditches have characteristic wetland attributes (e.g. hydric soils, aquatic plants and water regime) supports their use as a tool to transfer and transform contaminants and decrease the potential for effects to downstream receiving systems due to non-point source runoff (Moore et al., 2001). Agricultural ditches receive drainage water that may result from both irrigation and storm water runoff and therefore may contain elevated sediment, nutrient and pesticide loads.

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These ditches become not only an efficient means to promote drainage and reduce flooding on production acreage, but also a buffer between areas of intensive chemical use and ecosystems at potential risk. Moore et al. (2001) and Cooper et al. (2002) have shown that agricultural ditches can effectively transform pesticide-associated runoff from crop production.

Agricultural drainage ditches are integral components of the Mississippi Delta production landscape, and Moore et al. (2001) proposed these systems as comparable substitutes for edge-of-field constructed wetlands. Wetland vegetation continues to be investigated for its ability to partition pesticides from the water column (Moore et al., 2002; Schulz et al., 2003). Additional research is needed to quantify ditch ecosystem attributes (i.e. structural and functional components) that support the use of ditches as management practices for runoff.

This field study used the pyrethroid insecticide Asana XL® (esfenvalerate) [(S)-alpha-cyano-3-phenoxybenzyl(S)-2-(-4-chlorophenyl)-3-methylbutyrate]. Physical properties include low water solubility (0.002 mg/l at 25 °C) and high octanol/water coefficient ($K_{ow} = 10^{6.22}$). The reported aqueous half-life of esfenvalerate supported the use of short-term toxicity studies in determining sediment and aqueous partitioning effects on fish and invertebrates. This study attempted to elucidate the fate and effect of this insecticide during a simulated storm runoff event in a vegetated agricultural drainage ditch.

2. Methods and materials

A simulated storm event was conducted on a 650-m agricultural drainage ditch during the summer of 2000. Physical characteristics of the ditch included an average water width of 2.8 m, average water depth of 0.3 m and mean bed slope of 0.004. Concentrations of Asana XL® (esfenvalerate) were used to determine effects on standard test organisms exposed to ditch sediments and water. Native plant communities included 98% vegetative cover consisting of *Ludwigia peploides*, *Polygonum amphibium* and *Leersia oryzoides*.

A 110-l chamber was used to mix 86 ml esfenvalerate, 110 l of water, and 394 mg/kg of suspended sediment to simulate the storm event. The sediment was calculated from an estimated 0.394 g of dry sediment/l of runoff, and was used to simulate suspended sediment typically found in field runoff (Smith et al., 2002). A 7.6-cm diameter PVC pipe containing drilled holes along a 2.0-m length dispersed this suspension, and the remainder of the water used to simulate the runoff was simultaneously fed to the diffuser pipe via another route. Esfenvalerate was applied at the recommended field application rate with a simulated 0.64 cm event on a 20.23-ha agricultural field, which resulted in 12 870 l of water to simulate the runoff. One hour following the initiation of the simulated event, velocity was measured at 0.04 m/s, at three sampling locations with a Marsh-McBirney flowmeter. Sampling sites within the ditch mesocosm included an unamended (reference) upstream site (−10 m), injection point release site (0 m), and four affected downstream sites (20, 80, 200 and 600 m).

Aqueous grab samples were collected prior to exposure for background measurements of chemistry and toxicity at −10 and 600 m. Water from all sites was collected at 0.5-, 3- and 24-h post-application. Additionally, aqueous samples were collected from 0-, 20-, 80- and 200-m sites 28-d post-application. Following collection in amber glass bottles, the addition of ethyl acetate and potassium chloride facilitated removal of esfenvalerate for pesticide analysis. A composite of sediment from −10 through 100 m and a single grab sample from 600 m were collected prior to application and sediment samples were collected from all sites at 3- and 24-h post-application. Sediments from 0, 20 and 80 m were collected at 28-d post-application, and from 0 and 20 m at 56-d post-exposure. Sediments collected from the ditch mesocosm prior to chemical exposure were characterized for particle size fraction and percent volatile solids according to Gee and Bauder (1986). Sediment and aqueous samples collected in the field were placed on ice and delivered to the testing laboratory within six hours.

Standard test organisms, *Ceriodaphnia dubia* and *Pimphales promelas*, were utilized to determine acute toxicity to aqueous samples following methods outlined by US EPA (1993). Likewise, chemical analyses were conducted on unfiltered water, sediments and plants to determine pesticide concentrations throughout the 56-d period as described by Bennett et al. (2000), Moore et al. (2001) and Cooper et al. (2002). Esfenvalerate was analyzed by gas chromatograph equipped with a 30-m DB-1MS column. A multi-level calibration procedure was used with standards and was updated every ninth sample. Limits of detection (LOD) for esfenvalerate in water, sediments, and plants were 0.001 μg/l, 0.01 μg/kg, and 0.01 μg/kg, respectively; additionally, the limit of quantitation (LOQ) for esfenvalerate in water was 0.01 μg/l. Mean extraction efficiencies based on fortified samples, were >90% for water, sediment, and plants.

Acute sediment tests were conducted using solid phase, 10-d toxicity assays to determine relative inhibition of survival and growth in exposed *Chironomus tentans* (US EPA, 1994). A static flow-through system was used to renew overlying water in test chambers twice daily. Additionally, organisms in each test chamber were fed 1 ml of Tetramin® solution (4 g/l) daily during the 10 d. Overlying water quality was measured at regular intervals in randomly selected test chambers for each of the six field sites, which included temperature (°C), dissolved oxygen (mg/l), conductivity (μS/cm) and pH (APHA, 1998). Results from the toxicity assays were

statistically analyzed using Toxcalc® (version 5.0.25). A 48-h LC50 was calculated using measured esfenvalerate concentrations and corresponding partial and total response from aqueous test organisms. All LC50 determinations and related confidence intervals were determined using Probit analysis (Hamilton et al., 1977). All data were tested using $\alpha = 0.05$ and the normality assumption was tested using Shapiro–Wilk's test, and Steel's Many-One Rank test to determine significance in survival.

3. Results

Measured concentrations of esfenvalerate in water samples generally decreased as runoff progressed through downstream ditch sites (Table 1). Aqueous esfenvalerate was present from the injection point (0 m) to the 20-m site after 0.5-h exposure. Three hours after application, the highest aqueous pesticide concentration was measured in samples from the injection point (97.924 μg/l) while the lowest (1.064 μg/l) was measured in water collected from the 80-m site. The highest detectable aqueous pesticide concentration at 24 h was measured in water from the 80-m site (0.222 μg/l). After 28 d, esfenvalerate was detected only in water from the 200-m site (0.119 μg/l). Aqueous concentrations sufficient to cause significant acute mortality in *C. dubia* and *P. promelas* included those measured at 0 and 20 m at the 0.5-h sampling, and 0, 20, and 80 m at the 3-h sampling. The lowest measured aqueous pesticide concentration (0.222 μg/l) eliciting a response (45% survival) from *C. dubia* was measured at the 80-m site 24-h post-application, but did not cause decreased survival of

Table 1
Survival (±1 SD) of test organisms exposed to water from ditch mesocosm sites following esfenvalerate application on August 3, 2000 (measured esfenvalerate concentrations are furnished for each site)

Site	Survival (%)		Aqueous esfenvalerate[a] (μg/l)
	C. dubia	*P. promelas*	
0.5-h post-application			
Injection point (0 m)	0 ± 0[b]	0 ± 0[b]	29.430
20 m	0 ± 0[b]	0 ± 0[b]	8.326
80 m	95 ± 10	95 ± 10	nd
200 m	95 ± 10	92.5 ± 5	ns
600 m	95 ± 10	100 ± 0	ns
Control	100 ± 0	97.5 ± 5	ns
3-h post-application			
Injection point (0 m)	0 ± 0[b]	0 ± 0[b]	97.924
20 m	0 ± 0[b]	0 ± 0[b]	5.301
80 m	0 ± 0[b]	17.5 ± 15[b]	1.064
200 m	80 ± 0	95 ± 5.8	nd
600 m	100 ± 0	97.5 ± 5	nd
Control	100 ± 0	97.5 ± 5	ns
24-h post-application			
Injection point (0 m)	85 ± 10	97.5 ± 5	nd
20 m	80 ± 23	77.5 ± 38.6	0.099
80 m	45 ± 44[b]	95 ± 5.8	0.222
200 m	95 ± 10	72.5 ± 31	0.078
600 m	100 ± 0	97.5 ± 5	0.061
Control	100 ± 0	97.5 ± 5	ns
28-d post-application			
Injection point (0 m)	100 ± 0	92.5 ± 9.6	nd
20 m	100 ± 0	95 ± 5.8	nd
80 m	95 ± 10	60 ± 25.8[b]	nd
200 m	100 ± 0	85 ± 5.8	0.119
600 m	100 ± 0	97.5 ± 5	nd
Control	100 ± 0	90 ± 12.6	ns

nd = not detected (<0.001 μg/l).
ns = not sampled for GC analysis.
[a] Analyses performed on unfiltered aqueous samples.
[b] Significantly different from control.

P. promelas. Although aqueous esfenvalerate was below analytical detection after 28 d from the 80-m site, water from this site reduced survival of exposed *P. promelas* to 60% of control survival. Calculated 48-h LC50s for *C. dubia* and *P. promelas* were 0.215 μg/l (95% CI = 0.155–0.300 μg/l) and 0.616 μg/l (95% CI = 0.502–0.756 μg/l), respectively.

Particles in ditch sediments were predominantly silt (95%) with very little sand (4%) and clay (1%), containing 4.2% volatile solids. Pesticide concentrations measured in sediment samples obtained 3-h post-exposure ranged from 25.51 μg/kg at the injection site to 5.62 μg/kg in sediment from the 80-m site (Table 2). Sediment samples from all sites had detectable pesticide levels ranging from 0.26 to 104.30 μg/kg, 24 h after exposure. Measured pesticide concentrations were highest after 28 d at the injection point (450.01 μg/kg) and after 56 d (278.98 g/kg). *C. tentans* had partial survival upon exposure to samples collected from the 0-m site throughout the 56-d sampling. Sublethal responses (e.g. *C. tentans* growth) were also significantly reduced upon exposure to sediment samples collected at 24 h and 56 d from the injection point. Sediments collected from downstream sites elicited no other measurable response in *C. tentans*.

The highest pesticide levels in plant tissue were measured at 3-h post-exposure, 20 m from the point of injection (2010.34 μg/kg) and remained high through 80 m (1510.47 μg/kg) (Table 3). Pesticide levels were detected in plant samples collected at the 80-m site through 56-d post-application (21.81 μg/kg).

Table 3
Measured pesticide concentrations from plant materials extracted from ditch mesocosm sites following esfenvalerate application on August 3, 2000

Site	Esfenvalerate (μg/kg)			
	3 h	24 h	28 d	56 d
20 m	2010.34	701.67	189.35	ns
80 m	1510.47	577.94	53.03	21.81
200 m	2.35	2.79	nd	nd
600 m	ns	8.09	nd	6.35

ns = not sampled.
nd = not detected (<0.01 μg/kg LOD).

Table 2
Survival and measured growth (±1 SD) of *C. tentans* exposed to sediment samples from ditch mesocosm sites following esfenvalerate application on August 3, 2000 (measured esfenvalerate concentrations are furnished for each site)

Site	*Chironomus tentans*		Sediment esfenvalerate (μg/kg)
	Survival (%)	Growth (mg)	
3-h post-application			
Injection point (0 m)	25.0 ± 16.0[a]	0.88 ± 0.66[b]	25.51
20 m	65.0 ± 16.0	1.46 ± 0.64	13.13
80 m	87.5 ± 14.8	1.79 ± 0.50	5.62
200 m	93.8 ± 11.3	2.00 ± 0.65	ns
Control	83.3 ± 24.2	1.59 ± 0.29	ns
24-h post-application			
Injection point (0 m)	16.3 ± 7.4[a]	0.43 ± 0.27[a,b]	104.30
20 m	70.0 ± 25.1	1.28 ± 0.71	3.07
80 m	78.9 ± 27.5	1.48 ± 0.54	1.04
200 m	76.3 ± 19.2	1.71 ± 0.80	0.32
600 m	80.0 ± 36.6	1.17 ± 0.08	0.26
Control	83.3 ± 24.2	1.59 ± 0.29	ns
28-d post-application			
Injection point (0 m)	10.0 ± 10.0[a]	1.02 ± 0.65[b]	450.01
20 m	75.7 ± 16.2	1.65 ± 0.62	5.55
80 m	87.1 ± 9.5	2.07 ± 0.64	0.45
Control	87.5 ± 10.6	2.19 ± 0.38	ns
56-d post-application			
Injection point (0 m)	6.3 ± 7.4[a]	0.23 ± 0.17[a,b]	278.98
20 m	83.8 ± 11.9	1.99 ± 0.39	37.06
Control	87.5 ± 10.6	2.19 ± 0.38	ns

ns = not sampled for GC analysis.
[a] Significantly different from control.
[b] Evaluation of growth differences in this case is limited by small sample size related to the observed mortality.

4. Discussion

Although esfenvalerate dissipated from the water column quickly, the lowest-observed-adverse-effect concentration (LOAEC) to *C. dubia* was established at 0.222 μg/l. These results are similar to mesocosm studies by Fairchild et al. (1992), in which reported macroinvertebrate sensitivity to esfenvalerate was as low as 0.25 μg/l, and Giddings et al. (2001), who reported cladocera LOAEC of 0.310 μg/l for esfenvalerate. While no reported 48-h LC50 is found in literature for *C. dubia*, the calculated value of 0.215 μg/l from this study is comparable to the 0.22–0.27 μg/l range of reported 48-h LC50s for *D. magna* (Fairchild et al., 1992; Giddings et al., 2001). The LOAEC in this study for *P. promelas* (1.064 μg/l) was also comparable to the 0.880 μg/l reported by Giddings et al. (2001). Additionally, the 48-h LC50 calculated in the present study (0.616 μg/l) for the fathead minnow falls within the reported values of 0.34 μg/l for technical grade esfenvalerate and 0.93–1.13 μg/l for formulated esfenvalerate (Bradbury et al., 1987; Giddings et al., 2001).

The observed significant responses measured in *C. tentans* exposed to sediment from the injection point were expected, given the 25.51–450.01 μg/l esfenvalerate levels measured from this site. However, since Giddings et al. (2001) and Samsøe-Petersen et al. (2001) reported effects levels for *C. tentans* and *C. riparius* as 0.66 and 1.44 μg/l, respectively, the observed responses would be expected. Since no significantly reduced responses were measured at downstream sites, esfenvalerate in this field setting was less bioavailable than reported in other studies. Heinis and Knuth (1992) report that 51.0–97.7% of esfenvalerate may be sorbed to the top 1-cm layer of sediment, but uneven distribution of pesticides may occur in eroded sediments (Ghadiri and Rose, 1991). Therefore, laboratory derived LC50s for *Chironomus* may not offer comparable responses exhibited in actual field settings.

Transfer of esfenvalerate within the matrices of the ditch mesocosm was apparent as the measured aqueous esfenvalerate levels decreased from the point of injection and spatially throughout the ditch. With the absence of significant rainfall limiting runoff from adjacent agricultural fields throughout the duration of the exposure, rapid transfer onto plants and sediments was aided by increased hydrologic residence time and evident from both analytical and biological testing. Samsøe-Petersen et al. (2001) observed increased esfenvalerate adsorption to pond sediment with low suspended sediment in the water column, therefore the inclusion of sediment-spiked runoff offered an element of realism in the simulated exposure.

Although pesticide concentration in agricultural runoff is influenced by the hydrophobicity (K_{ow}) of the chemical, mechanical sheering of soil-bound pesticides by intense rainfall releases these chemicals into the water column, increasing field runoff (Ghadiri and Rose, 1991). Schulz et al. (1998) observed that measurable input of pesticides into surface waters may often occur with less water-soluble particle associated forms. Pesticide concentrations have been reported to be greater in eroded sediment than the original soil due to uneven distribution of organic matter and sorption within soil aggregates (Ghadiri and Rose, 1991). Whether aqueous or sediment bound, such runoff-related agricultural contaminants may be treated with a variety of suggested methods which include a series of wetland compartments, sheet flow through grassed buffer strips entering wetlands and edge-of-field conveyance systems serving as wetland substitutes (Brix and Schlerup, 1989; De Laney, 1995; Kadlec and Knight, 1996; Moore et al., 2000). As an essential component of these systems, macrophytes offer adsorption capabilities to hydrophobic pesticides, partitioning them from the water column and causing sedimentation of suspended particles (Mitsch and Gosselink, 2000). Sedimentation of particles from the water column is accomplished through increased substrate and reduced water velocity (Gregg and Rose, 1982; Madsen and Warncke, 1983; Watson, 1987).

Although little is known about the process of transfer onto aquatic plants and the potential metabolism by plants (Hand et al., 2001), a strong tendency of pyrethroids to be adsorbed to solid matter has been described by numerous authors (Hill, 1989; Zhou et al., 1995; Samsøe-Petersen et al., 2001; Maund et al., 2002). Macrophyte adsorption results in decreased esfenvalerate half-life from 15–90 to 14–28 d (EXTOXNET, 2003). Cooper et al. (2002) reported field half-lives of esfenvalerate in this same vegetated ditch as 0.12, 9 and 1.3 d for water, sediment and plants, respectively, illustrating the mitigation of the pesticide via macrophyte adsorption.

Aquatic macrophytes not only removed esfenvalerate from the water column through sorption, but also contributed additional organic matter to sediments, resulting in higher ditch sorptive capacity for hydrophobic pesticides. The spatial reduction of esfenvalerate due to mitigation by macrophytes and sediment resulted in reduced aquatic toxicity beyond the 200-m site. After 24 h of the exposure, acute toxicity to *P. promelas* was mitigated at all sampling sites, however, partial toxicity to *C. dubia* remained in water samples collected from the 80-m site (45% survival) at 24 h. No acute toxicity to *C. dubia* was noted in water from sampling sites after 28 d. A decrease in survival of *P. promelas* upon exposure to water from the 80-m site at 28 d was most likely not a response to aqueous esfenvalerate since the analytical measurements from this sample were below detectable limits.

Moore et al. (2000) demonstrated that between 100 and 280 m of a vegetated ditch were necessary for

effective mitigation of atrazine from runoff, while Cooper et al. (2002) determined that aqueous esfenvalerate could decrease to 0.1% of the initial exposure within 510 m of a vegetated ditch. Using standard test organisms, a minimum spatial distance of 200 m of vegetated conveyance structure was necessary to mitigate the toxic effects of esfenvalerate in the present agricultural runoff simulation. This investigation further illustrated the ability of sediments and vegetation in drainage ditches to serve as substrates for the dynamic conversion and transformation of stored and transported chemical mixtures from agricultural runoff.

Agricultural drainage ditches should be recognized and assessed for the most useful attributes that facilitate mitigation of pesticides and removal of runoff-associated contaminants. Greater emphasis might be placed on optimal spatial aspects and selective vegetation of these structures. Previous studies have supported the benefits of vegetated drainage ditches for mitigation of pesticides (Moore et al., 2001; Cooper et al., 2002). With appropriate management practices, mitigation of agricultural associated contaminants can be realized through existing vegetated drainage ditches thereby reducing the potential risk to downstream receiving systems.

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